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I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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Dated

4th February 2000



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# Request for grant of a patent

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The Patent Office

Cardiff Road  
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25 JAN 1999

1. Your reference

2. Patent ap

9901617.2

(The Patent Office will fill in ...)

25 JAN 1999

3. Full name, address and postcode of the or of each applicant (underline all surnames)

PETER FREDERICK WILDE  
91 FRONT STREET  
COWERBY THIRSK UK-407 1JP

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

NOT

6285712001

4. Title of the invention

IMPROVED MEANS FOR EXTRACTING FIXED AND MINERAL OILS FROM NATURAL RESOURCES

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

FS1/77  
3016109

Dibb Lupton Alsop  
Fountain Precincts  
Balm Green  
Sheffield  
S1 1RZ

Patents ADP number (if you know it)

7074917001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)

Date of filing  
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

- a) any applicant named in part 3 is not an inventor, or
  - b) there is an inventor who is not named as an applicant, or
  - c) any named applicant is a corporate body.
- See note (d))

**Patents Form 1/77**

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form

Description

10/

Claim(s)

NONE

Abstract

NONE

Drawing(s)

1+1-10

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11.

I/We request the grant of a patent on the basis of this application.

Signature

*G. H. Hinde*

Date 25/01/99

12. Name and daytime telephone number of person to contact in the United Kingdom

01 845 523 452

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DRAFT PATENT SPECIFICATION - JANUARY 13th 1999

IMPROVED MEANS FOR EXTRACTING FIXED AND MINERAL OILS  
FROM NATURAL RESOURCES

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January 13th 1999

The term "Fixed Oil" is usually used to describe oils of vegetable or animal origin which are not volatile oils. They routinely comprise natural mixtures of mono-, di- and tri-glycerides, fatty acids, sterols (and their esters) and natural waxes.

The term "Essential Oil" is usually used to describe those volatile oils of low molecular weight which incorporate the fragrance and flavour components derived from materials of natural origin.

"Mineral Oil" is a term usually used to describe petrochemical oils often derived from below ground level, which are normally mixtures of aliphatic and aromatic hydrocarbons of a very wide variety of chain length and molecular weight. They are often the sources of lubricating and fuel oils

In a previous patent specification (GBP 2,276,392), we described the remarkable properties of 1,1,1,2 - tetrafluoroethane (HFC 134a or R 134a) as a very appropriate solvent for the extraction of fragrant and aromatic essential oils from natural sources.

One very special feature of HFC 134a is that it is a very poor solvent for fixed oils and is thereby only capable of extracting very high quality fragrant and aromatic essential oils because it will not dissolve the fixed oils frequently associated with these components in the natural raw material.

Furthermore, it has been known to those familiar with the industry, that HFC 134a (which was developed in the late 1980's as a refrigerant intended to replace the environmentally unacceptable R12 - dichloro difluoromethane) is so poor a solvent that it was not adequately miscible or soluble in the mineral oils traditionally used as lubricants in refrigeration compressors.

This problem was so severe, in fact, that the international chemical industry was forced to synthesise completely new families of lubricants for the refrigeration compressors in systems in which it was intended that HFC 134a was to be used as the refrigerant.

HFC 134a is therefore regarded as a "very poor solvent" from conventional stand-points.

The preparation of bulk commodity "fixed oils" for culinary, cosmetic, food, pharmaceutical etc. use, frequently from seeds and nuts such as corn (maize), sunflower, grape pips, rape seeds, olive pits, oil palm nuts, sesame seeds, 'evening primrose' seeds, cocoa beans, copra (dried coconut flesh) etc., is normally carried out in the first instance by a pressing procedure.

The seeds or other raw materials are mechanically disrupted and then the oils is squeezed out of the disrupted seed bio-mass in some form of filter press. Hydraulic, screw and continuous cavitation screw presses are well known internationally as means of expelling such oils. The oil obtained by such pressing (in the case of olive oil, for instance) is referred to in product for retail sale as "virgin" or "extra virgin" olive oil.

Such presses however, are only able to expel and remove a proportion of the fixed oils from the "pressed cake".

The remaining oil in the "cake" may be allowed to remain there. Such "oil cake" is widely traded as animal food. However, in some cases (for example soya, evening primrose etc.), it would be economically foolish to discard the cake at this stage and steps are taken to obtain more oils from the cake by means of solvent extraction.

Frequently, the oil cake is stirred or otherwise brought into contact with a solvent such as hexane in which the fixed oil dissolves. In the past, benzene, dichloromethane and other undesirable chemical solvents have been employed for this purpose. The solution so formed, of fixed oil in the solvent, is filtered and the solvent is evaporated, leaving further oil available for sale. To achieve optimum economics, the cake may be "rinsed" several times with fresh solvent in order to remove the final traces of oil from it.

The cake may then be sold for inclusion in animal food.

Steam injection into the oil so formed, is frequently used as a means of lowering the final residues of hexane from the oil.

However, it is readily appreciated by all concerned that a proportion of residual solvent is still present and detectable in the oil derived by such processes.

The disadvantages of the process of hexane extraction of "oil cake" are many.

First, the solvent is expensive and losses are always encountered.

Secondly, the solvent is highly flammable and the fire hazard is always present in processing plants employing this solvent extraction procedure. Such fires have hitherto destroyed production facilities, killed or maimed the operators and caused damage to

surrounding property - quite apart from the inevitable economic loss of and disruption in production.

Thirdly, the lost solvent is almost always lost as a vapour. Such vapour (being hydrocarbon in nature) is a "VOC" (volatile organic compound) which has a photochemical ozone generating potential such that it is responsible, in the presence of bright sunlight at sea level, for the production of ozone, which is itself associated with the development of asthma and bronchitis and other breathing disorders in children in urban environments where many of these extraction plants would normally be situated.

Fourthly, the residual solvent inevitably present in the finished products from such processes (and intended for public consumption) may be toxic.

Fifthly, a major element of the cost of such solvent extraction processes is that of evaporation of the solvent from the hexane solution of the oil and its recovery by condensation.

HENCE IMPROVEMENTS TO THE PRESENT MEANS FOR THE EXTRACTION OF FIXED AND MINERAL OILS WOULD COMPRISE :

1. the elimination or reduction in the losses of solvent during such processes, by virtue of the use of a sealed system,
2. the use of a non-flammable solvent,
3. the use of a solvent which is not environmentally damaging and does not have a photochemical ozone generating potential,
4. the reduction in or absence of any toxic solvent residues in the final product,
5. the elimination of or reduction in the need to evaporate (and then condense) large quantities of solvents



The improvement we have made to these traditional processes addresses many of the disadvantages listed above and provides a means for approaching the ideals set out in 1 - 5 (above) and comprises the following:

HFC 134a, though a very poor solvent for fixed and mineral oils at low temperature is a very much better solvent at elevated temperature. At 40 degrees Celsius for example, cocoa butter (a fixed oil) dissolves in HFC 134a to an appreciable, useful and substantial extent.

Example 1:

A sample of 20 grams of roasted and finely ground cocoa beans was placed in a sealable container furnished with a closeable valve and capable of containing and withstanding pressures of 20 BarG. The in/outlet valve of the vessel was equipped with a filter to retain ground-up bio-mass within the vessel. 50 grams of HFC 134a was introduced into the vessel and the vessel sealed. A slurry was formed between the cocoa bean solids and the HFC 134a

A second (empty) similar vessel was prepared and the two vessels were connected by means of their inlet / outlet valves (which were both closed).

The two connected vessels, one containing the slurry and the other empty, were placed in an oven until the temperature of the contents rose to 50 degrees Celsius.

When the two vessels had warmed up to 50 degrees Celsius, the valves were opened so that the warm "solution" was able to pass from the vessel containing the bio-mass to the empty vessel. The valves were then closed.

The transfer and collection of the clear warm solution of cocoa butter in HFC 134a was readily accomplished via the filters. No bio-mass contaminated the clear solution.

Both vessels were allowed to cool.

Upon cooling, it was observed that cocoa butter had precipitated out of solution as a flocculent white precipitate.

Furthermore, due to the difference between the specific gravity of the "oil" (usually substantially lower than 1.00) and the solvent (substantially greater than 1.2) the precipitate was seen to rise to the surface of the (now cold) HFC 134a solvent leaving a clear layer of "solvent" below it. Further precipitation of cocoa butter solids could be encouraged by refrigeration of the vessels.

This characteristic facilitates the harvesting of the HFC 134a "solvent layer" (either by decantation or following further filtration). The clear cold solvent layer could now be returned to the vessel containing the original ground cocoa bean bio-mass.

Further warming caused more cocoa butter to be extracted and dissolved from the beans into the solvent and slowly over a period in which this cycle was repeated several times, a substantial amount of cocoa butter concentrated into the second vessel, leaving the roasted and ground cocoa beans in the first vessel largely devoid of cocoa butter.

This manual procedure, though highly effective, was somewhat tedious to carry out.

In fact the whole process can be built into a continuous operation. Simple laboratory scale equipment was built to study and develop this technology (see diagram) :

Two glass vessels A and B (such as are sold by Supelco and referred to as "Michel Miller glass columns (for high performance liquid chromatography)") were equipped with "shut-off" valves and coupled together via tubing as shown whereby below vessel A the tubing was in the form of a coil sitting in a bath of liquid which could be heated and maintained at a pre-selected temperature.

Vessel A was equipped with an internal filter at both ends whereas vessel B was equipped with a filter only at the lower end.

The second vessel B was surrounded by coils containing a flow of cooling liquid and the outside of the coils was insulated.

The circuit was furnished with an inlet (and outlet) valve for solvent. During operation of the equipment, this valve was coupled to a "header solvent storage bulk tank" which could be used to both fill and empty the system of solvent and keep it "topped up" during operation.

At the top of vessel B, a valve is fitted to facilitate the harvesting of oil when this becomes necessary.

The operation of the equipment may be described as follows:

- 1 Vessel A (which has removable end caps) is charged with the material from which oil is to be extracted (usually in the form of a finely divided particulate solid). The end caps are then replaced. The vessel is inserted into the remainder of the equipment.

2. The equipment (now fully sealed) is then fully charged with solvent from the bulk solvent storage tank (which remains connected to the equipment throughout the

operation). Air is allowed to escape from the equipment via judicious opening of the top valve.

3. The water bath is then filled with water and the hot plate turned on.

4. Cold liquid is circulated round the cooling coils causing the temperature of the second vessel B (and its contents) to cool.

As the temperature of the water in the water bath rises, so does the temperature of solvent in the tube below vessel A. This of course causes the hot liquid solvent to rise through the contents of the vessel A which are restrained inside vessel A by the filters top and bottom.

The liquid so displaced upwards is replaced by cold liquid falling through vessel B.

The entire liquid circuit thus becomes mobile and circulating. As hot liquid passes up through the contents of vessel A oil is extracted from this material. As the solution enters the top of vessel B it is cooled and its solute (the oil) precipitates out of solution.

Because the oil is lighter than the solvent, it floats to the top of vessel B and collects there as an ever expanding layer.

When it is considered that sufficient oil has been extracted, all the valves are closed (except valves C and D) and the valve at the top of vessel B (valve E) is opened and the contents decanted into a bottle for further study.

The system may be emptied by allowing solvent to drain out of valve X into a suitable container for recovery by evaporation and re-cycling.

It will be immediately apparent to one versed in the art, that this process is capable of producing oil without any evaporative step. As evaporation of the solvent is one of the major costs involved in more traditional methods of extraction, this constitutes a major advance and improvement in the extraction of such oils.

Furthermore, the solvent is neither flammable, nor toxic, nor environmentally damaging and (in normal operation) is never released into the environment.

This elegant and simple system of extraction relies on the two simple principles that :

- a. Fixed and mineral oils are more soluble in hot solvent than in cold solvent (in which they are barely soluble at all) and
- b. They are lighter than the solvent and hence tend to float upon it.

#### Example 2.

A sample of North Sea drilling mud comprised a highly acidic moist powder of finely ground mineral particles, water and oil. In the past, this mud has been jettisoned from the drill platform directly into the sea. This practice is coming under close scrutiny by those concerned for the environment.

If a process could be provided which recovered some of the contaminating oil from such slurries, disposal into the sea may be allowed to continue. The economics of such a process would obviously be crucial to its adoption, however, the value of the oil recovered could help off-set the inevitable on-costs of treatment.

100 grams of North Sea drilling mud was loaded into a vessel such as that described as vessel A in the diagram. The entire system was then assembled and sealed and filled with solvent [in this case a mixture of HFC 134a (90 % w/w) and butane (10 % w/w)].

The temperature of the contents of vessel A was allowed to rise as the contents of vessel B were cooled. Solvent circulated quickly around the system and a pale yellow oil began to accumulate at the top of vessel B.

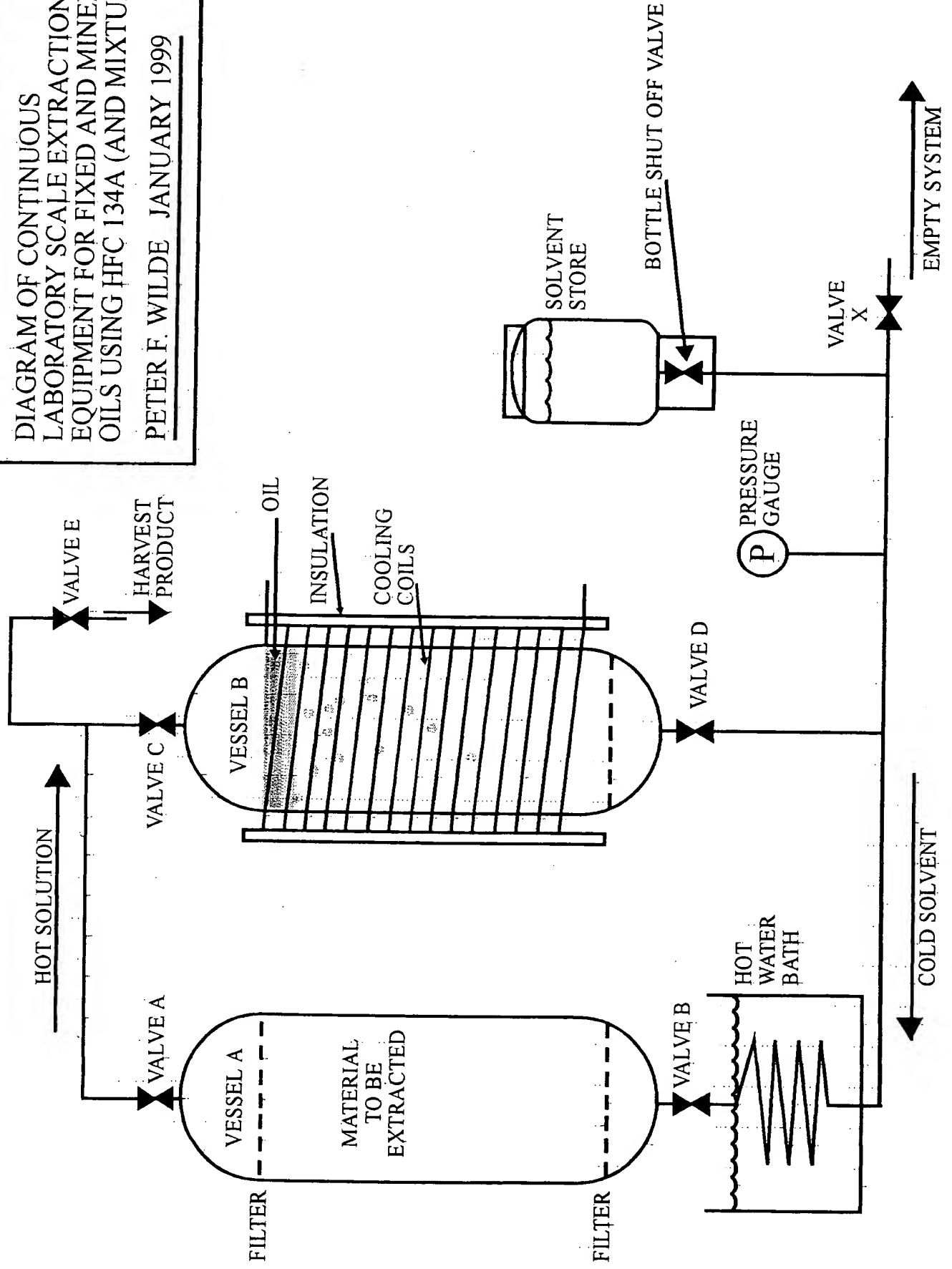
After 20 minutes of operation at equilibrium conditions (after stable temperatures had been achieved), the system was shut down. All valves (except valves D and C and the bottle shut-off valve) were closed. Upon opening of valve E, solvent emerged and was collected in a bottle. Opening of valve C also caused solvent to emerge into the bottle. In so doing, the layer of oil in vessel B was observed to rise. As oil emerged through valve E, it was collected into a second sample bottle and subjected to analysis.

A small quantity of solvent was seen to "boil-off" the oil sample. On the larger scale, this solvent could have been recovered and re-used.

The oil was found to be of excellent (light) and saleable quality.

# DIAGRAM OF CONTINUOUS LABORATORY SCALE EXTRACTION EQUIPMENT FOR FIXED AND MINERAL OILS USING HFC 134A (AND MIXTURES)

PETER F. WILDE JANUARY 1999



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